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UTILITY PATENT APPLICATION TRANSMITTAL

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37 CFR §1.53(b))

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Sir:

Transmitted herewith for filing is the patent
application of:

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RADDATZ

For: USE OF POLYALPHAOLEFINS (PAO)
DERIVED FROM DODECENE OR
TETRADECENE TO IMPROVE THERMAL
STABILITY IN ENGINE OIL IN AN INTERNAL
COMBUSTION ENGINE

Case Docket No. 98 400 204.8
Chevron Corporation
Law Department - Patent and Licensing Unit
P. O. Box 6006
San Ramon, CA 94583-0806

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Enclosed are:

- ☒ Specification [Total Pages = 28].
- ☒ An Assignment of the invention to Chevron Chemical SA, a corporation of France.
- ☐ A certified copy of a _____ application.
- ☒ Drawing(s) [Total Sheets = 1].
- ☒ Combined Oath or Declaration and Power of Attorney [Total Pages = 3].
 - ☐ Newly executed (original or copy)
 - ☐ Copy from a prior application (37 CFR §1.63(d)) (for continuation/divisional)
 - ☐ **DELETION OF INVENTOR(S)**
Signed statement attached deleting inventor(s) named in the prior application, see 37 CFR 1.63(d)(2) and 1.33(b).
- ☒ Please amend the specification by adding the following as the first sentence: This application is a continuation-in-part of Serial No. PCT/IB99/00141, filed January 27, 1999 which claims priority from EPO application 98 400.8 filed January 30, 1998.
- ☐ Information Disclosure Statement.
- ☒ If a CONTINUING APPLICATION, check appropriate box and supply the requisite information:
 - ☐ Continuation ☐ Divisional ☒ Continuation-in-part (CIP) of prior application No: PCT/IB99/00141 filed January 27, 1999 which claims priority from EPO Application 98 400 204.8 filed January 30, 1998.

The filing fee has been calculated as shown below:

	(Col. 1)	(Col. 2)
For	No. Filed	No. Extra
Basic Application Fee		
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JWAmbrosius:pik
Enclosures

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July 24, 2000

135030

1 BE IT KNOWN, that We, FRANK STUNNENBERG, a citizen of the
2 Netherlands, resident of Diemen, the Netherlands; PERLA DUCHESNE, a
3 citizen of France, resident of Notre Dame de Gravenchon, France; and
4 JUERGEN H. RADDATZ, a citizen of Germany, resident of Darmstadt-
5 Eberstadt, Germany, have invented new and useful improvements in

6 **USE OF POLYALPHAOLEFINS (PAO) DERIVED FROM DODECENE OR**
7 **TETRADECENE TO IMPROVE THERMAL STABILITY IN ENGINE OIL IN**
8 **AN INTERNAL COMBUSTION ENGINE**

9

10 CROSS REFERENCE TO RELATED APPLICATIONS

11 The present application is a continuation-in-part of PCT Application
12 1B/99/00141 filed January 27, 1999 which claims priority from EPO
13 Application 98 400 204.8 filed January 30, 1998.

14

1 **USE OF POLYALPHAOLEFINS (PAO) DERIVED FROM DODECENE OR**
2 **TETRADECENE TO IMPROVE THERMAL STABILITY IN ENGINE OIL IN**
3 **AN INTERNAL COMBUSTION ENGINE**

4 FIELD OF THE INVENTION

5 The present invention relates to compositions of automotive engine oils using
6 synthetic poly alpha olefins derived from 1-dodecene or 1-tetradecene, to
7 improve engine oil performance, as demonstrated by the severe Volkswagen
8 T-4, Volkswagen TDI, and Sequence IIIE tests.

9 BACKGROUND OF THE INVENTION

10 Today's automobiles tend to have smaller, more demanding engines
11 operating at higher temperatures. Thus, the engine oil has to function in an
12 increasingly severe environment while meeting fuel economy demands.
13 Besides changes in the additive package, increasingly synthetic base oils are
14 being used instead of conventional mineral oils. Of the synthetic oils, poly
15 alpha olefins (PAO) are among the most popular.

16 PAO is manufactured by the oligomerization of linear alpha olefins followed
17 by hydrogenation to remove unsaturated bonds and fractionation to obtain the
18 desired product slate. 1-decene is the most commonly used alpha olefin in
19 the manufacture of PAO, but 1-dodecene and 1-tetradecene can also be
20 used. PAO's are commonly categorized by the numbers denoting the
21 approximate viscosity in centistokes of the PAO at 100°C. It is known that
22 PAO 2, PAO 2.5, PAO 4, PAO 5, PAO 6, PAO 7, PAO 8, PAO 9 and PAO 10
23 and combinations thereof can be used in engine oils. The most common of
24 these are PAO 4, PAO 6 and PAO 8.

25 Conventionally, base oils of lubricating viscosity used in motor oil
26 compositions may be mineral oil or synthetic oils of viscosity suitable for use
27 in the crankcase of an internal combustion engine. Crankcase base oils

1 ordinarily have a viscosity of about 1300 cSt at 0°F (-18°C) to 24 cSt at 210°F
2 (99°C). The base oils may be derived from synthetic or natural sources.
3 Mineral oil for use as the base oil in this invention includes paraffinic,
4 naphthenic and other oils that are ordinarily used in lubricating oil
5 compositions. Synthetic oils include both hydrocarbon synthetic oils and
6 synthetic esters.

7 Although the common 1-decene based PAO 4, 6 and 8 offer better
8 performance than mineral oil based engine oils, they encounter difficulties
9 when subjected to the severe PV 1449, CEC L-78-T-96 and Sequence IIIE
10 tests. The PV 1449 and Sequence IIIE tests evaluate fully formulated engine
11 oils with respect to high temperature oxidative stability and piston deposits.
12 The CEC L-78-T-96 test evaluates fully formulated engine oils with respect to
13 piston cleanliness and piston ring sticking. The PV 1449 and CEC L-78-T-96
14 tests will be referred to hereinafter as the Volkswagen T-4 and TDI engine
15 tests, respectively.

16 It has been found to be difficult to blend an engine oil of the desired 0W30
17 viscosity grade based on PAO 4 and 6 that successfully completes the TDI
18 test. Repeatedly, it was found that too low oil pressure caused the engine to
19 fail from 2 to 8 hours before the end of the test. In the T-4 test, it was found
20 that the increase in engine oil viscosity resulting in engine failure during the
21 test was related to oil oxidation stability and volatility. To pass the T-4 test, it
22 was found that the PAO 4/6 based engine oil requires large quantities of
23 expensive anti-oxidants. The other way to obtain PAO 4/6 based oil which
24 passes the T-4 test is to use an expensive fully synthetic oil.

25 The Volkswagen T-4 and TDI tests have recently become an important
26 measure of engine lubrication oil quality under very severe conditions. The
27 Sequence IIIE test is analogous to a T-4 test but is specifically developed for
28 U.S. built engines. The T-4 and Sequence IIIE tests are for gasoline engines
29 and the TDI test is for diesel engines. They replicate the severe engine
30 conditions put on motor lubrication oil by sustained, very high speed driving,

1 as on the German Autobahn. What is needed is a PAO based oil which is
2 able to successfully complete severe engine tests such as the Volkswagen
3 T-4 and TDI tests and the Sequence IIIE test without having to use large
4 quantities of anti-oxidants or a fully synthetic oil.

5 Surprisingly, it has been found that lubrication oils based on a feed consisting
6 of 1-dodecene or 1-tetradecene, and that have approximate viscosities at
7 100°C of from 3.5 to 8.5 centistokes, successfully pass the T-4 and TDI tests
8 with PAO based oil weight percentages much lower than previously achieved.
9 This represents a major development in the search for an economical
10 lubrication oil that is well suited for modern driving conditions.

11 SUMMARY OF THE INVENTION

12 In its broadest aspect the present invention relates to a base oil composition
13 suitable for use in an engine oil which comprises a mixture of trimer and
14 higher oligomers derived from an alpha olefin feed consisting essentially of
15 either 1-dodecene or 1-tetradecene wherein said oligomer mixture contains
16 less than 2 weight percent of combined monomer and dimer. When used in
17 this specification the phrase "consisting essentially of either 1-dodecene or 1-
18 tetradecene" refers to a feed which contains at least 85% by weight of 1-
19 dodecene or 1-tetradecene. In the preferred embodiment of the invention the
20 base oil composition will consist essentially of only the trimer and higher
21 oligomers of either 1-dodecene or 1-tetradecene. The term "oligomer
22 mixture" as used herein is intended to mean a mixture of the different
23 oligomers of either dodecene or tetradecene. It is not intended to mean a
24 mixture of oligomers derived from alpha olefins other than dodecene or
25 tetradecene.

26 The present invention also relates to the use of PAO oil as a base oil, or as a
27 component of a base oil, in an engine oil for the purpose of improving the
28 high temperature stability wherein the PAO oil comprises a mixture of trimer
29 and higher oligomers derived from an olefin feed consisting of either 1-

1 dodecene or 1-tetradecene wherein said oligomer mixture contains less than
2 2 weight percent of combined monomer and dimer.

3 In another embodiment, the present invention relates to the use of the PAO
4 derived from 1-dodecene or 1-tetradecene as a base oil, or a component of a
5 base oil, in an engine oil comprised of said base oil, in addition to dispersants,
6 detergents, oxidation inhibitors, foam inhibitors, anti-wear agents and at least
7 one viscosity index improver, for the purpose of improving the high
8 temperature stability of the engine oil to at least the point at which the engine
9 oil is able to pass the VW T-4, VW TDI, or Sequence IIIE tests. Preferably,
10 the base oil comprises between 15 to 85 weight percent of the engine oil and
11 at least 15 weight percent of the base oil consists of the PAO derived from
12 1-dodecene or 1-tetradecene.

13 The PAO derived from 1-dodecene or 1-tetradecene, as used in the present
14 invention, preferably will have a viscosity at 100°C of between about 3.5
15 centistokes to about 9.5 centistokes. Particularly preferred for use in
16 manufacturing base oils of the present invention are those PAO's having a
17 viscosity at 100°C of approximately 5 centistokes, approximately 6
18 centistokes, or approximately 7 centistokes, i.e, PAO 5, PAO 6, or PAO 7.
19 Especially preferred for use in the present invention are PAO 5 and PAO 7.
20 The viscosity of the PAO will depend upon the relative percentage of the
21 various oligomers present in the product. In general, the higher the
22 percentage of higher molecular weight oligomers, the higher the viscosity of
23 the PAO. Thus for example, in the case of dodecene, PAO 5 would have a
24 higher percentage of trimer present than PAO 6 or PAO 7. PAO 7 would
25 have a higher percentage of tetramer or higher oligomers than PAO 5 or PAO
26 6. The different viscosity PAO's are readily separated by distillation to yield
27 the desired oligomer cut.

28 As used in this disclosure the words "comprises" or "comprising" is intended
29 as an open-ended transition meaning the inclusion of the named elements,
30 but not necessarily excluding other unnamed elements. The phrase "consists

1 essentially of" or "consisting essentially of" is intended to mean the exclusion
2 of other elements of any essential significance to the composition. When
3 specifically referring to the feed composition the phrase "consisting
4 essentially of either 1-dodecene or 1-tetradecene" refers to a feed which
5 contains at least 85% by weight of 1-dodecene or 1-tetradecene. The
6 phrases "consisting of" or "consists of" are intended as a transition meaning
7 the exclusion of all but the recited elements with the exception of only minor
8 traces of impurities.

9 Unless explicitly stated otherwise, all percentages in this specification refer to
10 percent by weight.

11 BRIEF DESCRIPTION OF THE DRAWINGS

12 In order to assist the understanding of this invention, reference will now be
13 made to the appended drawings. The drawings are exemplary only, and
14 should not be construed as limiting the invention.

15 Figure 1 is a graph comparing the absolute and relative T-4 viscosity
16 increases in PAO 6 and PAO 5/7 based motor oil in an experiment the
17 conditions of which are described in Example 5.

18 Figure 2 is a graph comparing the absolute and relative T-4 viscosity
19 increases in PAO 4, PAO 5 and PAO 6 based motor oil in an experiment the
20 conditions of which are described in Example 6.

21 DETAILED DESCRIPTION OF THE INVENTION

22 As discussed above, the present invention is concerned with improving the
23 thermal stability, oxidative stability, and volatility characteristics of engine oil
24 by using a base oil composition prepared from PAO derived from the
25 oligomers of 1-dodecene or 1-tetradecene. The amount of monomer and
26 dimer present in the PAO used for preparing the base oil of the present
27 invention should comprise no more than 2.0 weight percent. Preferably the

1 PAO should consist only of trimers or higher oligomers of 1-dodecene or 1-
2 tetradecene. Surprisingly, it has been found that PAO 5 and PAO 7 derived
3 from 1-dodecene or 1-tetradecene offer superior thermal stability, oxidation
4 stability, and volatility characteristics when used as a base oil as compared to
5 PAO 4 and PAO 6 derived from decene. As the examples below show, such
6 improved oxidation stability is found in both gasoline (T-4) and diesel (TDI)
7 engines (especially direct injection diesels). Furthermore, the superior
8 oxidation stability qualities are shown in both fully synthetic as well as
9 semi-synthetic engine oils, which are a mixture of PAO's and mineral oils.
10 PAO 5/7 when used as a base oil has also been shown to be superior over
11 PAO 4/6/8 in PSA TU3M high temperature gasoline tests and Sequence IIIE
12 high temperature oxidation tests.

13 As discussed above, it is essential that the alpha olefin feed used to prepare
14 the PAO which in turn is used to prepare the base oil be a relatively pure feed
15 of either 1-dodecene or 1-tetradecene, i.e., containing no more than 15% by
16 weight of other alphaolefins. Mixtures containing more than 15% by weight of
17 other alpha olefins are not suitable as a feedstock in preparing the PAO used
18 in the practice of the present invention. More preferably the feed will contain
19 less than 10% by weight of other alphaolefins. In addition, the PAO should
20 never contain more than 2 weight percent of dimer or residual monomer.
21 Accordingly, the carbon chains of the PAO used to prepare the base oils of
22 the present invention will contain multiples of either 12 or 14 carbon atoms,
23 such as, in the case of dodecene, 36, 48, 60 carbon atoms, etc. or in the case
24 of tetradecene, 42, 56, 70 carbon atoms, etc. This molecular consistency has
25 been found to impart some very desirable properties to the base oil prepared
26 from the PAO, as for example, the ability to pass the very stringent VW T-4
27 test.

28 Generally for base oils used to prepare 0W-20-50 SAE viscosity grade engine
29 oils, the PAO will comprise from 50% to 85% by weight of the base oil. For
30 base oils used to prepare 5W-20-50 SAE viscosity grade engine oils, the PAO
31 will comprise from 15% to 50% by weight of the base oil. For base oils used

1 to prepare 10W-20-50 SAE viscosity grade engine oils, the PAO will comprise
2 from 5 % to 35% by weight of the base oil.

3 In addition to the base oil derived from the PAO of the present invention,
4 commercial engine oils typically contain various other additives, such as
5 dispersants, detergents, anti-wear agents, oxidation inhibitors, foam
6 inhibitors, and viscosity index improvers. These other additives used in the
7 formulation of a typical engine oil are discussed below.

8 ADDITIVE COMPONENTS

9 The following additive components represent examples of some components
10 that can be favorably employed in preparing engine oils of the present
11 invention. These examples of additives are provided to illustrate the present
12 invention, but they are not intended to limit it:

13 (1) Metal detergents: sulfurized or unsulfurized alkyl or alkenyl phenates,
14 alkyl or alkenyl aromatic sulfonates, sulfurized or unsulfurized metal
15 salts of multi-hydroxy alkyl or alkenyl aromatic compounds, alkyl or
16 alkenyl hydroxy aromatic sulfonates, sulfurized or unsulfurized alkyl or
17 alkenyl naphthenates, metal salts of alkanoic acids, metal salts of an
18 alkyl or alkenyl multi-acid, metal salts of an alkyl salicylic acid,
19 carboxylates, overbased detergents and chemical and physical mixtures
20 thereof.

21 (2) Ashless dispersants: alkenyl succinimides, alkenyl succinimides
22 modified with other organic compounds, and alkenyl succinimides
23 modified with boric acid, alkenyl succinic ester.

24 (3) Oxidation inhibitors:

25 (a) Phenol type oxidation inhibitors: 4,4'-methylenebis (2,6-di-tert-
26 butylphenol), 4,4'-bis(2,6-di-tert-butylphenol), 4,4'-bis(2-methyl-6-
27 tert-butylphenol), 2,2'-(methylenebis (4-methyl-6-tert-butyl-phenol),

1 4,4'-butylidenebis(3-methyl-6-tert-butylphenol),
2 4,4'-isopropylidenebis(2,6-di-tert-butylphenol), 2,2'-methylenebis(4-
3 methyl-6-nonylphenol), 2,2'-isobutylidene-bis(4,6-dimethylphenol),
4 2,2'-methylenebis(4-methyl-6-cyclohexylphenol), 2,6-di-tert-butyl-
5 4-methylphenol, 2,6-di-tert-butyl-4-ethylphenol, 2,4-dimethyl-6-tert-
6 butyl-phenol, 2,6-di-tert-4-(N.N' dimethylaminomethylphenol),
7 4,4'-thiobis(2-methyl-6-tert-butylphenol), 2,2'-thiobis(4-methyl-6-
8 tert-butylphenol), bis(3-methyl-4-hydroxy-5-tert-butylbenzyl)-sulfide,
9 and bis (3,5-di-tert-butyl-4-hydroxybenzyl).

10 (b) Diphenylamine type oxidation inhibitor: alkylated diphenylamine,
11 phenyl-I-naphthylamine, and alkylated I-naphthylamine.

12 (c) Other types: metal dithiocarbamate (e.g., zinc dithiocarbamate),
13 and methylenebis (dibutyldithiocarbamate).

14 (4) Rust inhibitors (Anti-rust agents):

15 (a) Nonionic polyoxyethylene surface active agents: polyoxyethylene
16 lauryl ether, polyoxyethylene higher alcohol ether, polyoxyethylene
17 nonylphenyl ether, polyoxyethylene octylphenyl ether,
18 polyoxyethylene octyl stearyl ether, polyoxyethylene oleyl ether,
19 polyoxyethylene sorbitol monostearate, polyoxyethylene sorbitol
20 mono-oleate, and polyethylene glycol monooleate.

21 (b) Other compounds: stearic acid and other fatty acids, dicarboxylic
22 acids, metal soaps, fatty acid amine salts, metal salts of heavy
23 sulfonic acid, partial carboxylic acid ester of polyhydric alcohol, and
24 phosphoric ester.

25 (5) Demulsifiers: addition product of alkylphenol and ethyleneoxide,
26 polyoxyethylene alkyl ether, and polyoxyethylene sorbitan ester.

- 1 (6) Extreme pressure agents (EP agents): zinc dithiophosphates, zinc
2 dithiocarbamates, zinc dialkyldithiophosphate (primary alkyl type &
3 secondary alkyl type), zinc diaryl dithiophosphate, sulfurized oils,
4 diphenyl sulfide, methyl trichlorostearate, chlorinated naphthalene,
5 fluoroalkylpolysiloxane, and lead naphthenate.
- 6 (7) Friction modifiers: fatty alcohol, fatty acid, amine, borated ester, and
7 other esters.
- 8 (8) Multifunctional additives: sulfurized oxymolybdenum dithiocarbamate,
9 sulfurized oxymolybdenum organo phosphoro dithioate, oxymolybdenum
10 monoglyceride, oxymolybdenum diethylate amide, amine-molybdenum
11 complex compound, and sulfur-containing molybdenum complex
12 compound.
- 13 (9) Viscosity index improvers: polymethacrylate type polymers, ethylene-
14 propylene copolymers, styrene-isoprene copolymers, hydrated styrene-
15 isoprene copolymers, polyisobutylene, and dispersant type viscosity
16 index improvers.
- 17 (10) Pour point depressants: polymethyl methacrylate.
- 18 (11) Foam Inhibitors: alkyl methacrylate polymers and dimethyl silicone
19 polymers.
- 20 In one embodiment, an engine lubricating oil composition would contain:
 - 21 (a) a major part of a base oil of lubricating viscosity, wherein the base oil
22 comprises 1-dodecene and/or 1-tetradecene-derived polyalphaolefins;
 - 23 (b) 0% to 20% of at least one ashless dispersant;
 - 24 (c) 0% to 30% of the detergent;

- 1 (d) 0% to 5% of at least one zinc dithiophosphate;
- 2 (e) 0% to 10% of at least one oxidation inhibitor;
- 3 (f) 0% to 1% of at least one foam inhibitor; and
- 4 (g) 0% to 20% of at least one viscosity index improver.

5 In a further embodiment of the present invention, an engine lubricating oil
6 composition is produced by blending a mixture of the above components.
7 The lubricating oil composition produced by that method might have a slightly
8 different composition than the initial mixture, because the components may
9 interact. The components can be blended in any order and can be blended
10 as combinations of components. In general, most engine oil compositions will
11 contain between 5% and 85% by weight of base oil.

12 A preferred engine oil composition of the present invention will include from 0
13 to about 20 weight percent of at least one ashless dispersant, from 0 to about
14 30 weight percent of detergent, from 0 to about 5 weight percent of at least
15 one anti-wear agent, from 0 to about 10 weight percent of at least one
16 oxidation inhibitor, from 0 to about 1 weight percent of at least one foam
17 inhibitor, and from 0 to about 20 weight percent of at least on viscosity
18 improver.

19 In addition to the compositions discussed above, preferred engine oil
20 compositions having a SAE viscosity grade of 0W20-40 are comprised of
21 from 15 to 85% of a base oil containing from 50 to 85% of PAO at least 15
22 weight percent of which is derived from 1-dodecene or 1-tetradecene
23 according to the present invention. Likewise, in the case of engine oil
24 compositions having a SAE viscosity grade of 5W20-40, the compositions are
25 preferably comprised of from 15 to 85 weight percent of a base oil containing
26 from 15 to 50 weight percent of PAO at least 15 weight percent of which is
27 derived from 1-dodecene or 1-tetradecene. For those engine oil compositions
28 having a SAE viscosity grade of 10W20-50 are comprised of from 15 to 85

1 weight percent of a base oil containing from 5 to 35 weight percent of PAO at
2 least 15 weight percent of which is derived from 1-dodecene or 1-
3 tetradecene.

4 ADDITIVE CONCENTRATES

5 Additive concentrates are also included within the scope of this invention.
6 The concentrates of this invention comprise the compounds or compound
7 mixtures of the present invention, with at least one of the additives disclosed
8 above. Typically, the concentrates contain sufficient organic diluent to make
9 them easy to handle during shipping and storage.

10 From 20% to 80% of the concentrate is organic diluent. Suitable organic
11 diluents which can be used include for example, solvent refined 100N, i.e.,
12 Cit-Con 100N, and hydrotreated 100N, i.e., RLOP 100N, and the like. The
13 organic diluent preferably has a viscosity of from about 1 to about 20 cSt at
14 100°C.

15 EXAMPLES

16 The invention will be further illustrated by the following examples, which set
17 forth particularly advantageous embodiments. While the Examples are
18 provided to illustrate the present invention, they are not intended to limit it.

19 Examples 1 through 4 cover bench test data obtained in the proprietary MAO
20 92 oxidation bench test. In this test, air is bubbled through an oil sample at
21 elevated temperature. The oil sample contains an oxidation catalyst. The
22 viscosity of the oil at 40°C is measured at regular intervals until 1000 cSt is
23 reached. The time to reach this value is a measure of the stability. The
24 longer the time, the better the oxidation stability. The MAO 92 oxidation test
25 has a repeatability of 7 hours. PAO 5 and 7 referred to in the following
26 examples are derived from 1-dodecene according to the present invention.
27 PAO 4, 6, and 8 are derived from 1-decene.

-12
EXAMPLE 1

A fully formulated engine oil was prepared, containing an additive package comprised of 6% dispersant, 71.5 mmol detergent, 15.5 mmol zinc dithiophosphate, 0.55% supplementary additives, 2.0% VII, 34.8% Esso 145N, 20.55% Esso 600N and 15% PAO 5 and 15% PAO 7. This oil was subjected to the MAO 92 oxidation test, the result being 125 hours.

COMPARATIVE EXAMPLE 2

As a comparison, a similar engine oil as described in Example 1 was prepared. However, the 15% PAO 5 and 15% PAO 7 were replaced by 30% PAO 6. The result of the oxidation test was only 100 hours.

EXAMPLE 3

The experiment of Example 1 was repeated using an additive package comprised of 6% dispersant, 71.5 mmol detergent, 15.5 mmol zinc dithiophosphate, 0.55% supplementary additives, 2.0% VII, 52% PAO 5 and 33.3% PAO 7. The result in the oxidation test is 162 hours.

COMPARATIVE EXAMPLE 4

As a comparison to Example 3, the PAO 5 and 7 were replaced by 11.1% PAO 4 and 74.2% PAO 6. The result in the oxidation test, 152 hours, was poor in comparison to the oil of Example 3.

EXAMPLE 5

The oils of Example 1 and Comparative Example 2 were subjected to the bench tests used to mimic the viscosity increase of the VW T-4 engine test. The lower the absolute and relative viscosity increase, the better the test result. As can be seen in Figure 1, the oil based on PAO 5/7 is far superior to the oil based on PAO 6.

-13
TABLE 1

Oil code	OIL 10	OIL 11
Additive package	AP7	AP7
PAO 5		15
PAO 6	30	
PAO 7		15
Calculated T-4 viscosity (cSt)	756.6	201.8
Calculated T-4 viscosity increase (%)	819.0	189.7

EXAMPLE 6

A fully formulated engine oil was prepared containing an additive package comprised of 6% dispersant, 87 mmol detergent, 19 mmol zinc dithiophosphate and 0.35% supplementary additives, 10.3% VII and 30% PAO 5, the balance made up by mineral base stock. Two similar engine oils were prepared but the 30% PAO 5 was replaced by 30% PAO 4 and 30% PAO 6, respectively. These three oils were subjected to the bench tests used to mimic the viscosity increase of the VW T-4 engine test. The lower the absolute and relative viscosity increase, the better the test result. As can be seen in Figure 2, the oil based on PAO 5 is far superior to the oils based on PAO 4 and PAO 6.

-14
TABLE 2

Oil code	OIL 13	OIL 12	OIL 14
Additive package	AP4	AP4	AP4
PAO 5	30		
PAO 4		30	
PAO 6			30
Calculated T-4 viscosity (cSt)	99.4	258.2	154.3
Calculated T-4 viscosity increase (%)	10.5	212	79.5

EXAMPLE 7

A fully formulated engine oil was prepared containing an additive package comprised of 6.5% dispersant, 98 mmol detergent, 5.5 mmol zinc dithiophosphate and 1.8% supplementary additives, 4.0% VI improver and the balance a 57.6/42.4 mixture of PAO 4 and PAO 6. This oil was run in the VW TDI engine. The test was aborted after 52 hours, i.e., 8 hours before reaching the end-of-test, as result of low oil pressure due to a lack of engine oil remaining in the sump.

A VW TDI test was conducted on a 1.9 liter turbo charged, intercooled DI diesel type engine. The engine tested has power of 81 kW at 4150 rpm's. There are 4 cylinders in the engine measuring 79.5 x 95.5 mm (b x s). EGR is not activated in the engine and the oil charge is 4.5 liters. The test procedure had a 5 hour run-in step, a 3 hour power curve step, and a 2 hour flushing step.

These steps were followed by a 60 hour cycling step which had two stages: stage 1, the idling stage; and stage 2, the full load stage. One cycle takes three hours and the cycle was repeated 20 times (20 x 3 hrs.). Further facts about the cycling stage are given in Table 3 below.

TABLE 3

CEC L-78-T-96 (TDI) Engine Test

Test Conditions

	Stage 1	Stage 2
Duration (minutes)	30	150
Speed (rpm)	Idle	4150
Oil Temperature (°C)	40	145
Coolant Temperature (°C)	30	90
Boost Air Temperature (°C)	30	60

COMPARATIVE EXAMPLE 8

As a comparison to Example 7, the PAO 4 and 6 were replaced by 8.6% PAO 5 and 91.4% PAO 7. The oil successfully completed the 60 hour VW TDI engine test.

EXAMPLE 9

T-4 bench tests and engine tests were performed on oil compositions containing various additives, including viscosity index improvers and various proportions of PAO 4, PAO 5, PAO 6, PAO 7, PAO 8 and mineral stock. Tables 4A through 4D show the T-4 bench test and engine test results as well as the MAO 92 results for the compositions. These results show the correlation between the engine test results and the bench test model for both the absolute viscosity at end-of-test (EOT) and also for the relative viscosity increase. Both are requirements for the T-4 test.

The Engine Test Conditions for conducting the VW T-4 test are given below in Table 4. The total test had a duration of 262 hours (10 hours run-in, + 2 hours power curve, + 2 hours flushing, + 48 x PNK cycles = 48 x 4 = 192 hrs, + 56 hrs N cycle → 262 hours). The test oil charge was 5 liters with no oil top-up allowed. Of the various test requirements, the limits on viscosity

- 1 increase are the most difficult to achieve. Both relative viscosity increase as
2 well as absolute viscosity increase at EOT are limited. The limits are as
3 follows: EOT Viscosity at 40°C <200 cSt.
4 EOT Viscosity increase <130%.

-17
TABLE 4A

Oil Code	OIL 1	OIL 2	OIL 3
Additive Package	AP1	AP 2	AP3
--dispersant (wt%)	n.a.	5	6.75
--detergent (mmol)	n.a.	84	70
-zinc dithiophosphate (mmol)	n.a.	18	18
-supplementary additives (wt%)	n.a.	1.6	0.93
VI Improver (%)	n.a.	4.7	10.5
VI Improver		polymethyl- acrylate type polymers (PMA)	ethylene propylene copolymers (OCP)
PAO 4	n.a.		
PAO 5	n.a.		
PAO 6	n.a.	62.1	25
PAO 7	n.a.		
PAO 8	n.a.	20	
Mineral Stock (%)	n.a.		50.6
Mineral Stock	n.a. full synth.		Group 1
TGA (°C)	336.8	342.5	312.5
MAO 92-visc. at 100 H (cSt)	69.3	125.9	180.1
MAO 92-visc. increase at 100 H (%)	-9.8	65.9	87.1
Calculated VW T-4 viscosity increase (cSt)	107.8	114.1	302.8
Calculated VW T-4 viscosity increase (%)	47.9	55.3	264.0
Act. T-4 visc. increase (cSt)	134.2	107.0	450.9
Act. T-4 visc. increase (%)	74.5	41.0	368.5

-18
TABLE 4B

1

Oil Code	OIL4	OIL5	OIL6
Additive Package	AP2	AP4	AP5
--dispersant (wt%)	5	6	6.5
--detergent (mmol)	84	87	98
-zinc dithiophosphate (mmol)	18	19	15.5
-supplementary additives (wt%)	1.6	0.35	1.8
VI Improver (%)	6.2	9	6.3
VI Improver	OCP	OCP	Styrene isoprene copolymers (Styr.-IP)
PAO 4			45.5
PAO 5			
PAO 6	21.8	23.5	13.1
PAO 7			
PAO 8			
Mineral Stock (%)	58.8	55	20
Mineral Stock	Group I	Group I	Group II
TGA (°C)	316.2	318.7	320
MAO 92-visc. at 100 H (cSt)	1344.6	190.9	74
MAO 92-visc. increase at 100 H (%)	1326.5	108.7	32.3
Calculated VW T-4 viscosity increase (cSt)	1017.4	277.2	197.3
Calculated VW T-4 viscosity increase (%)	971.1	236.2	182.7
Act. T-4 visc. increase (cSt)	Too viscous to measure	335.4	151.7
Act. T-4 visc. increase (%)		268.0	171.2

-19
TABLE 4C

1

Oil Code	OIL7	OIL8	OIL9
Additive Package	AP5	AP5	AP6
--dispersant (wt%)	6.5	6.5	6
--detergent (mmol)	98	98	93
-zinc dithiophosphate (mmol)	15.5	15.5	19
-supplementary additives (wt%)	1.8	1.8	1.6
VI Improver (%)	5.2	5.0	5.0
VI Improver	Styr.-IP	Styr.-IP	Styr.-IP
PAO 4	43	15.98	15.98
PAO 5		63.92	63.92
PAO 6	36.7		
PAO 7			
PAO 8			
Mineral Stock (%)			
Mineral Stock			
TGA (°C)	314	353	355
MAO 92-visc. at 100 H (cSt)	53.8	51.1	-25.4
MAO 92-visc. increase at 100 H (%)	-1.3	50.5	-25.3
Calculated VW T-4 viscosity increase (cSt)	215.5	12.9	-45.6
Calculated VW T-4 viscosity increase (%)	202.1	-22.4	-80.2
Act. T-4 visc. increase (cSt)	115.0		
Act. T-4 visc. increase (%)	108.0		

2

-20
TABLE 4D

Oil Code	OIL10	OIL11
Additive Package	AP7	AP7
--dispersant (wt%)	6	6
--detergent (mmol)	71.5	71.5
-zinc dithiophosphate (mmol)	15.5	15.5
-supplementary additives (wt%)	0.55	0.55
VI Improver (%)	2.0	2.0
VI Improver	OCP	OCP
PAO 4		
PAO 5		15
PAO 6	30	
PAO 7		15
PAO 8		
Mineral Stock (%)	55.3	55.3
Mineral Stock	Group I	Group I
TGA (°C)	310	325
MAO 92-visc. at 100 H (cSt)	880	122
MAO 92-visc. increase at 100 H (%)	1000	99.7
Calculated VW T-4 viscosity increase (cSt)	756.6	201.8
Calculated VW T-4 viscosity increase (%)	819.0	189.7
Act. T-4 visc. increase (cSt)		
Act. T-4 visc. increase (%)		

TABLE 5

VW PV 1449 ENGINE TEST (T-4)

Test Conditions

PNK Cycles	Max Power P	Max NO _x N	Cold Idling K	Max NO _x N
Duration	120 min	72 min	48 min	56 hrs
RPM	4300	4300	900	4300
Oil Sump Temp °C	133	130	40	130
Coolant Temp °C	100	100	30	100
Power kW	62	34	0	34
Torque Nm	140	75	0	75
Fuel Cons. kg/h	19.4	10.8	1.1	10.8
Exh. Gas Temp °C	820	763	292	763

EXAMPLE 10

Bench Test Thermal Gravimetric Analysis (TGA) of PAO 5 and 7

Bench test analysis was performed on four different samples of oil to find the TGA DPeak (i.e. the temperature at which the weight loss, due to both evaporation and thermal degradation, of the oil is the most important, which correlates with oil consumption). This test measures the weight variation of a sample as a function of temperature, under a nitrogen flow. At a certain temperature, defined as the DPeak, the weight loss is the most important. The exact DPeak value is determined as the maximum of the derivative curve. The repeatability of the TGA test is equal to 8°C. Table 6 shows the results which support the superiority of PAO 5 and 7 in a bench scale test.

TABLE 6

	Test 1	Test 2	Test 3	Test 4
Dispersant wt%	6.5	6.5	6	6
Detergent mmol	98	98	71.5	71.5
Zinc dithiophosphate mmol	15.5	15.5	15.5	15.5
Supplementary additives wt%	1.8	1.8	0.55	0.55
VII wt %	5.2	5.2	2.0	2.0
PAO 4/6 wt %	43/36.7			
PAO 4/5 wt %		15.98/63.92		
PAO 6 wt %			30	
PAO 5/7 wt %				30
Mineralstock wt %			55.3 Esso	55.3 Esso
TGA (°C)	314	353	310	325

-23
EXAMPLE 11

1
2 A fully formulated engine oil was prepared, containing 13.6% of an additive
3 package, 6.9% VI Improver, 10% ester and 35% PAO 5 and 34.5% PAO 7. A
4 Seq. IIIE test was run on this oil with a 1986 3.8 liter Buick V6 engine using
5 leaded gasoline. The initial oil fill is 5.3 liters. Total test duration is 64 hours.
6 The engine speed is 3000 rpm with a load of 50.6 kW. The oil temperature is
7 149°C. The results of the test were as follows:

8	— viscosity increase:	-11%
9	— time to 375% vis. incr.:	87.3 hours
10	— Aver. engine sludge:	9.7
11	— oil consumption, liter	0.67

12 As a comparison, a similar engine oil as described above was prepared.
13 However, the 35% PAO 5 and 34.5% PAO 7 were replaced by 69.5% PAO 6.
14 Again, a Seq. IIIE was run, resulting in:

15	— viscosity increase:	-1%
16	— time to 375% vis. incr.:	85.8 hours
17	— Aver. engine sludge:	9.6
18	— oil consumption, liter	1.14

19 The results show the superiority of PAO 5 and 7 over PAO 6 in the Seq. IIIE
20 test.

21 While the present invention has been described with reference to specific
22 embodiments, this application is intended to cover those various changes and
23 substitutions that may be made by those skilled in the art without departing
24 from the spirit and scope of the appended claims.

1 WHAT IS CLAIMED IS:

- 2 1. A base oil composition suitable for use in an engine oil which comprises
3 a mixture of trimer and higher oligomers derived from an alpha olefin
4 feed consisting essentially of either 1-dodecene or 1-tetradecene
5 wherein said oligomer mixture contains less than 2 weight percent of
6 combined monomer and dimer.
- 7 2. A base oil according to claim 1 wherein the base oil composition
8 consists essentially of only the trimer and higher oligomers of either 1-
9 dodecene or 1-tetradecene.
- 10 3. The base oil composition of claim 2 wherein the trimer and higher
11 oligomers are derived from 1-dodecene.
- 12 4. An engine oil comprising between 50% and 85% by weight of the base
13 oil of claim 1.
- 14 5. The engine oil of claim 4 further including from 0 to about 20 weight
15 percent of at least one ashless dispersant, from 0 to about 30 weight
16 percent of detergent, from 0 to about 5 weight percent of at least one
17 anti-wear agent, from 0 to about 10 weight percent of at least one
18 oxidation inhibitor, from 0 to about 1 weight percent of at least one foam
19 inhibitor, and from 0 to about 20 weight percent of at least one viscosity
20 improver.
- 21 6. An engine oil according to claim 5 having an SAE viscosity grade of
22 0W-20-40 comprising from 15 to 85% by weight of the base oil which is
23 comprised of from 50 to 85% by weight of PAO, wherein at least 15% by
24 weight of said PAO is derived from 1-dodecene or 1-tetradecene.
- 25 7. An engine oil according to claim 5 having an SAE viscosity grade of
26 5W-20-50 comprising from 15 to 85% by weight of the base oil which is

1 comprised of from 15 to 50% by weight of PAO, wherein at least 15% by
2 weight of said PAO is derived from 1-dodecene or 1-tetradecene.

3 8. An engine oil according to claim 5 having an SAE viscosity grade of
4 10W-20-50 comprising from 15 to 85% by weight of the base oil which is
5 comprised of from 5 to 35% by weight of PAO, wherein at least 15% by
6 weight of said PAO is derived from 1-dodecene or 1-tetradecene.

7 9. A method for improving the thermal stability, oxidative stability, and
8 volatility characteristics of engine oil which comprises using a base oil
9 comprised of PAO derived from a linear alpha olefin feed consisting
10 essentially of 1-dodecene or 1-tetradecene.

11 10. The method of claim 9 wherein the PAO has a viscosity at 100°C of
12 between about 3.5 to about 9.5 centistokes.

13 11. The method of claim 10 wherein the PAO has an approximate viscosity
14 at 100°C of 5 centistokes.

15 12. The method of claim 10 wherein the PAO has an approximate viscosity
16 at 100°C of 7 centistokes.

17 13. An engine oil able to pass the VW T-4, VW TDI, or Sequence IIIE tests
18 which comprises from about 5 to about 85 weight percent of a base oil,
19 from 0 to about 20 weight percent of at least one ashless dispersant,
20 from 0 to about 30 weight percent of detergent, from 0 to about 10
21 weight percent of at least one oxidation inhibitor, from 0 to about 1
22 weight percent of at least one foam inhibitor, and from 0 to about 20
23 weight percent of at least one viscosity improver, wherein the base oil
24 comprises a mixture of trimer and higher oligomers derived from an
25 alpha olefin feed consisting essentially of either 1-dodecene or 1-
26 tetradecene and wherein said oligomer mixture contains less than 2
27 weight percent of combined monomer and trimer.

- 1 14. The engine oil of claim 13 wherein the base oil consists of trimer and
- 2 higher oligomers derived from 1-dodecene.

CALCULATED VW T-4 VISCOSITY INCREASE

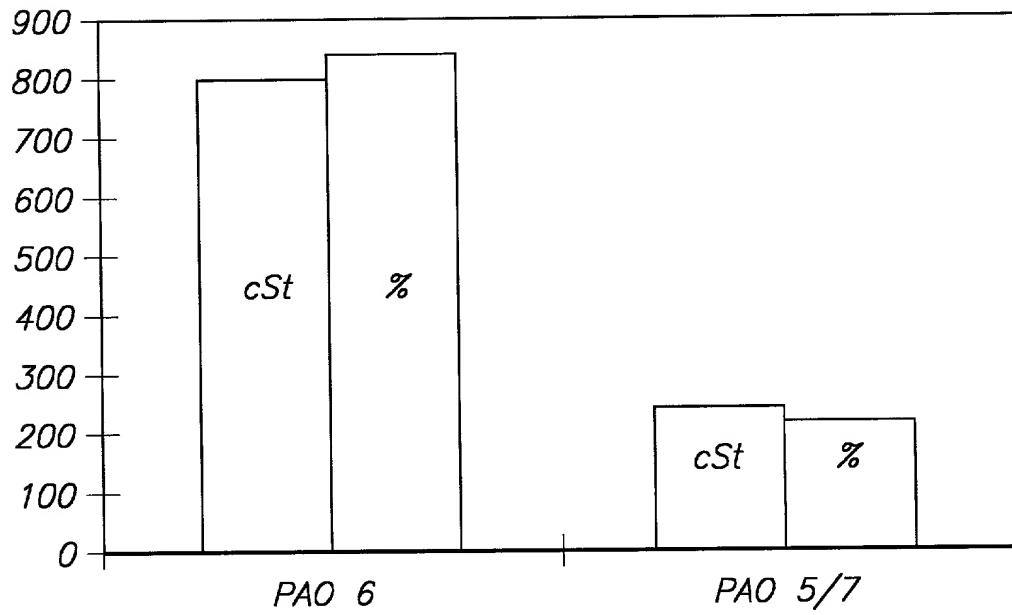


FIG. 1

CALCULATED VW T-4 VISCOSITY INCREASE

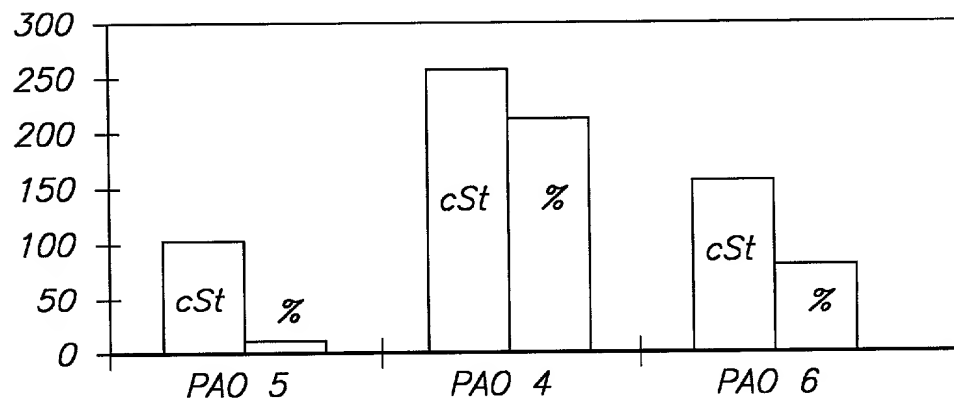


FIG. 2

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name;

I BELIEVE I AM THE ORIGINAL, FIRST, AND SOLE INVENTOR (if only one name is listed below) OR AN ORIGINAL, FIRST, AND JOINT INVENTOR (if more than one name is listed below) OF THE SUBJECT MATTER WHICH IS CLAIMED AND FOR WHICH A PATENT IS SOUGHT ON THE INVENTION

Entitled: **USE OF POLYALFAOLEFINS (PAO) DERIVED FROM 1-DODECENE OR 1-TETRADECENE TO IMPROVE THERMAL STABILITY IN ENGINE OIL IN AN INTERNAL COMBUSTION ENGINE**

the specification of which:

(check one) ☒ is attached hereto:

☐ was filed on as
Application Serial No.
and was amended on
(if applicable)

I HAVE REVIEWED AND UNDERSTAND THE CONTENTS OF THE ABOVE-IDENTIFIED SPECIFICATION, INCLUDING THE CLAIMS, AS AMENDED BY ANY AMENDMENT REFERRED TO ABOVE:

I ACKNOWLEDGE THE DUTY TO DISCLOSE INFORMATION WHICH IS MATERIAL TO THE PATENTABILITY OF THIS APPLICATION IN ACCORDANCE WITH TITLE 37, CODE OF FEDERAL REGULATIONS, Sec. 1.56(a) which states: "A patent by its very nature is affected with a public interest. The public interest is best served, and the most effective patent examination occurs when, at the time an application is being examined, the Office is aware of and evaluates the teachings of all information material to patentability. Each individual associated with the filing and prosecution of a patent application has a duty of candor and good faith in dealing with this Office, which includes a duty to disclose to the Office all information known to that individual to be material to patentability as defined in this section. The duty to disclose information exists with respect to each pending claim until the claim is cancelled or withdrawn from consideration, or the application becomes abandoned".

I hereby claim foreign priority benefits under Title 35, United States Code Sec. 119 and/or Sec. 365 of any foreign application(s) for patent or inventor's certificate as indicated below and have also identified below any foreign application for patent or inventor's certificate on this invention having a filing date before that of the application on which priority is claimed:

COUNTRY/INTERNATIONAL	APPLICATION NUMBER	DATE OF FILING (Day, Month, Year)	PRIORITY CLAIMED
European Patent Office	98 400 204.8	30 January 1998	Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>
PCT	IB/99/00141	27 January 1999	Yes <input checked="" type="checkbox"/> No <input type="checkbox"/>

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations § 1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

APPLICATION NUMBER	DATE OF FILING	STATUS

I HEREBY APPOINT THE FOLLOWING AS OUR ATTORNEYS WITH FULL POWER OF SUBSTITUTION TO PROSECUTE THIS APPLICATION AND TRANSACT ALL BUSINESS IN THE PATENT AND TRADEMARK OFFICE CONNECTED THEREWITH:

W. Keith Turner
James W. Ambrosius
Thomas G. DeJonghe

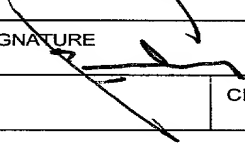
REGISTRATION NO.
26,816
27,705
24,467

ASSOCIATE POWER OF
ATTORNEY ATTACHED
Yes ☐ No ☒

SEND CORRESPONDENCE TO:

Chevron Corporation
Law Department
Patent and Licensing Unit
P.O. Box 6006
San Ramon, CA 94583-0806

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FULL NAME OF SOLE OR FIRST JOINT INVENTOR FRANK STUNNENBERG	SIGNATURE 	DATE July 31, 2000
RESIDENCE Pater Pirestraat 26, 1111 Kr Diemen, The Netherlands	CITIZENSHIP The Netherlands	
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF SECOND JOINT INVENTOR, IF ANY PERLA DUCHESNE	SIGNATURE	DATE
RESIDENCE 8, Rue President R. Andre, 76330, Notre-dame de Gravenchon, France	CITIZENSHIP France	
POST OFFICE ADDRESS (Same as above)		

☒ Please see attached continuation page for additional inventors.

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name;

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the specification of which:

(check one) ☒ is attached hereto:

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APPLICATION NUMBER	DATE OF FILING	STATUS

I HEREBY APPOINT THE FOLLOWING AS OUR ATTORNEYS WITH FULL POWER OF SUBSTITUTION TO PROSECUTE THIS APPLICATION AND TRANSACT ALL BUSINESS IN THE PATENT AND TRADEMARK OFFICE CONNECTED THEREWITH:

W. Keith Turner
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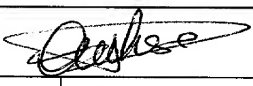
REGISTRATION NO.
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ASSOCIATE POWER OF
ATTORNEY ATTACHED
Yes ☐ No ☒

SEND CORRESPONDENCE TO:

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P.O. Box 6006
San Ramon, CA 94583-0806

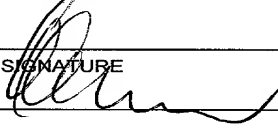
I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FULL NAME OF SOLE OR FIRST JOINT INVENTOR FRANK STUNNENBERG	SIGNATURE	DATE
RESIDENCE Pater Pirestraat 26, 1111 Kr Diemen, The Netherlands	CITIZENSHIP The Netherlands	
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF SECOND JOINT INVENTOR, IF ANY PERLA DUCHESNE	SIGNATURE 	DATE 11/7/2000
RESIDENCE 8, Rue President R. Andre, 76330, Notre-dame de Gravenchon, France	CITIZENSHIP France	
POST OFFICE ADDRESS (Same as above)		

☒ Please see attached continuation page for additional inventors.

COMBINED DECLARATION AND POWER OF ATTORNEY CONTINUATION PAGE FOR ADDITIONAL INVENTORS

(This page must be attached to a completed Combined Declaration and
Power of Attorney before Signing.)

FULL NAME OF THIRD JOINT INVENTOR, IF ANY JURGEN H. RADDATZ	SIGNATURE 	DATE 10th July 2002
RESIDENCE Heidelberger Landstrasse 112a, 64297 Darmstadt-Eberstadt, Germany		CITIZENSHIP Germany
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF FOURTH JOINT INVENTOR, IF ANY	SIGNATURE	DATE
RESIDENCE		CITIZENSHIP
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF FIFTH JOINT INVENTOR, IF ANY	SIGNATURE	DATE
RESIDENCE		CITIZENSHIP
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF SIXTH JOINT INVENTOR, IF ANY	SIGNATURE	DATE
RESIDENCE		CITIZENSHIP
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF SEVENTH JOINT INVENTOR, IF ANY	SIGNATURE	DATE
RESIDENCE		CITIZENSHIP
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF EIGHTH JOINT INVENTOR, IF ANY	SIGNATURE	DATE
RESIDENCE		CITIZENSHIP
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF NINTH JOINT INVENTOR, IF ANY	SIGNATURE	DATE
RESIDENCE		CITIZENSHIP
POST OFFICE ADDRESS (Same as above)		
FULL NAME OF TENTH JOINT INVENTOR, IF ANY	SIGNATURE	DATE
RESIDENCE		CITIZENSHIP
POST OFFICE ADDRESS (Same as above)		